# Nuclear Magnetic Resonance (NMR) Handbook

**United States Department of Agriculture**

Grain Inspection, Packers and Stockyards Administration

Federal Grain Inspection Service

Washington, DC

May 2003

United States Department of Agriculture
Grain Inspection, Packers and Stockyards Administration
Packers and Stockyards Administration

# Program Handbook

5-12-03

## Nuclear Magnetic Resonance Testing for Sunflower Seed Oil

Foreword

The Nuclear Magnetic Resonance (NMR) Handbook has been established to provide procedures for official personnel to follow for the testing and certification of sunflower seed oil using the NMR method.

All official inspection personnel authorized or licensed to perform NMR testing shall reference this handbook for procedures.

This handbook supersedes the FGIS Nuclear Magnetic Resonance (NMR) Handbook dated April 3, 1997.

***/s/ David Orr***

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# CHAPTER 1

## GENERAL INFORMATION

## 1.1 SCOPE

Testing sunflower seed for oil content as "official criteria" is authorized under Section 7(b) of the United States Grain Standards Act (USGSA), as amended. All official sunflower seed oil analysis under the USGSA is performed in accordance with procedures prescribed in this handbook by authorized Federal Grain Inspection Service (FGIS) employees or licensed personnel employed by delegated/designated agencies.

The continuous wave and pulsed low resolution Nuclear Magnetic Resonance (NMR) instruments are the only approved instruments for official sunflower seed oil determination. The NMR method is based on the principle of activating hydrogen atoms in the oil using electromagnetic radiation and a magnet. The NMR reading is a measure of the number of activated hydrogen atoms. Technical Services Division (TSD) will use the petroleum ether oil extraction method (AOCS method number Ai 3-75) as a standard to which the NMR instruments are calibrated and referenced.

This handbook establishes procedures for officially determining and certificating oil content of sunflower seed, monitoring the accuracy of official sunflower seed oil results, and maintaining sunflower seed oil equipment accuracy.

## 1.2 DEFINITIONS

**Check Samples** - Sunflower seed samples tested by the Board of Appeals and Review and distributed to all specified service points for monitoring the uniformity of field results.

**Collaborative Study** - A study designed to compare NMR oil values determined by different laboratories.

**Constituent** - Compounds for which an analysis is made in a product, i.e., oil in sunflower seed.

**Continuous Wave NMR** - A technique used for determining the oil content of a sunflower seed sample by measuring the number of electromagnetically activated hydrogen atoms present in liquid oil. Continuous Wave (CW) instruments continuously apply external radio frequency energy to samples while simultaneously scanning the magnetic field. The signal is recorded by measuring the absorption of radiation by the nuclei at their resonant frequencies.

**Correlation** - The interdependency of one variable on another, i.e., the amount of oil extractable from a sample using petroleum ether and NMR response.

**Monitor Samples** - Sunflower seed samples randomly selected from the market which are analyzed and compared to a monitoring office.

**NMR Response** - A measure of the number of activated hydrogen atoms within a magnetic field.

**Oil** - A mixture of a glyceride ester of fatty acids widely occurring in organic tissues that are liquid at room temperature.

**Petroleum Ether Oil Extraction** - A chemical determination of percent oil in a sample.

**Pulsed NMR** - A technique used for determining the oil content of a sunflower seed sample by measuring the number of electromagnetically activated hydrogen atoms present in liquid oil. Pulsed NMR instruments apply external radio frequency energy to samples as short pulses lasting a few microseconds. The pulses simultaneously excite all of the nuclei in a sample and the signal (called a free induction decay) is measured after the pulse. All modern NMR instruments use pulsed techniques.

**Reference Value** - An oil value determined by TSD for each of the Sunflower Seed Standard samples and Sunflower Seed Check samples.

**Slope** - The degree of slant of the regression line.

**Specified Service Point** - A city, town, or other location specified by an agency for the performance of official inspection or Class X or Class Y weighing services.

**Sunflower Seed Standard (SSS)** - Dried and sealed sunflower seed samples with established weights and NMR oil values. SSS are prepared by TSD and distributed to specified service points to calibrate NMR instruments.

**Tuning Sample (TS)** - A sample (sunflower seed oil or relaxed mineral oil) giving a signal large enough to tune the NMR instrument.

## 1.3 RESPONSIBILITIES

The general responsibilities for the sunflower seed oil testing program are as follows:

a. Responsibilities of the Technical Services Division.

(1) Maintain the standard reference petroleum ether oil extraction laboratory for FGIS and create calibrations for approved NMR instruments used for official NMR oil testing.

(2) Establish the official oil content of all Sunflower Seed Standards (SSS).

(3) Provide SSS to field offices and all service points providing official NMR sunflower seed oil determination.

(4) Monitor the capability of the official sunflower seed oil testing program.

(5) When necessary, review sunflower seed oil analysis procedures at FGIS field offices and specified service points.

(6) Recommend corrective and follow-up action when problems are detected.

(7) Provide technical support and training to official personnel in matters relating to oil analysis.

(8) Initiate and/or conduct and report collaborative and/or special studies as needed.

(9) When needed, perform calibration studies and make recommendations.

(10) Provide Board appeal inspection for sunflower seed oil testing.

(11) Issue certificates and assess fees for Board appeal inspection service.

b. Responsibilities of FGIS Field Office Managers.

(1) Coordinate and maintain the sunflower seed NMR oil testing program within the circuit.

(2) At domestic locations, perform or make arrangements to perform appeal inspections for sunflower seed oil testing services within the field office circuit.

(3) At export locations, provide original, reinspection, and appeal sunflower seed oil testing services in areas not assigned to an official agency.

(4) Forward file samples for Board appeal testing services to TSD.

(5) Select and forward samples for monitoring to the Grand Forks Field Office. The Grand Forks Field Office will send their samples selected for monitoring to TSD.

(6) Monitor the performance of specified service points within the circuit.

(7) Review oil testing procedures at specified service points within the circuit.

(8) Immediately inform TSD of problems detected in the circuit and initiate corrective and follow-up action.

(9) Provide technical support and training to official inspection personnel.

(10) Assist TSD in conducting collaborative and/or special studies.

c. Responsibilities of Official Agency Managers/State Cooperators.

(1) Coordinate and maintain a sunflower seed oil testing program within the assigned geographic area.

(2) Perform original and reinspection NMR sunflower seed oil testing services within the assigned geographic area and forward file samples for appeal sunflower seed oil testing services to the FGIS Grand Forks Field Office.

(3) Select and forward samples for monitoring to the Grand Forks Field Office.

(4) Routinely review oil analysis procedures at specified service points within the assigned geographic area.

(5) Permit only official personnel who are trained and licensed for sunflower seed oil testing to perform such activities.

(6) Provide technical support and training to licensed inspection personnel within the assigned geographic area.

(7) Assist TSD and/or FGIS monitoring field office in conducting collaborative and/or special studies.

(8) Inform the monitoring field office manager and TSD of problems detected within the assigned geographic area and initiate corrective and follow-up action.

# CHAPTER 2

## NMR OIL TESTING EQUIPMENT

## 2.1 PURPOSE

To ensure that the FGIS sunflower oil program is applied uniformly by all specified service points, that only approved continuous wave or pulsed Nuclear Magnetic Resonance (NMR) equipment and procedures are used, that equipment maintenance and testing schedules are followed, and that laboratory setups conform to FGIS specifications.

## 2.2 APPROVED EQUIPMENT

The following equipment is approved for official NMR sunflower oil determinations.

**Aluminum Dishes** - Aluminum dishes with lids, 89 millimeters (mm) in diameter and 51 mm deep with closely fitted slip-on cover.

**Balance** - An approved electronic balance with 0.01 gram precision and RS-232 output (Mettler PM-200 or equivalent).

**Bottle Brushes** - Bottle brushes for cleaning NMR sample tubes.

**Desiccating Cabinet** - An airtight cabinet which can be equipped with a desiccant material to maintain a very low relative humidity environment.

**Desiccant** - Silica gel for drying, indicating, 6-16 mesh.

**Electronic Power Conditioner (Optional)** - An electronic power line conditioner (Sola model No. EPC 150-60, Tripp Lite model LS-604 or equivalent).

**Forced Air Convection Oven** - A forced air convection oven (Blue M Model OV-490A-2, or equivalent).

**NMR Instruments** - The Newport Analyzer Models Mark III-A and Oxford 4000 model (continuous wave wide-line NMR) equipped with type 10 magnet (Watson 10) and 150 milliliter (ml) magnet coil assembly. (Oxford Instruments Inc., 130-a Baker Avenue Extension, Concord, MA 01742, phone 1-800-447-4717).

**Note: The Mark III-A model is approved for official use but is not in service at the current time. Therefore, the handbook does not contain step-by-step procedures for instrument operation.**

Oxford Instruments pulsed NMR models MQA 6005 and MQA 7005 equipped with 5 Mhz magnet and 150 ml magnet coil assembly.

Universal Maran Ultra pulsed NMR equipped with 5 Mhz magnet and 150 ml magnet coil assembly. (Universal Systems, 29500 Aurora Road, Unit 16, Solon, OH 44139, phone 1-440-349-3210).

Bruker minispec mq7.5 pulsed NMR equipped with 7.5 Mhz magnet and 150 ml magnet coil assembly. (Bruker Canada Ltd., Milton, Ontario L9T 1Y6, Canada, phone 1-905-876-4641).

**NMR Sample Tubes** - NMR sample tubes for 150 mL NMR magnet coil assembly.

**Rubber Stoppers** - Rubber stopper for NMR sample tube, size No. 10.

**Thermometers** - Thermometer with 1° to 51°C scale and 0.1°C divisions.

## 2.3 DISCLAIMER CLAUSE

The mention of firm names or trade products does not imply that they are endorsed or recommended by the U.S. Department of Agriculture over other firms or similar products not mentioned. Except for the NMR instrument, equivalent equipment may be used in place of the items listed.

## 2.4 OIL TESTING FACILITIES

Equipment location and environmental factors can affect the performance of NMR sunflower seed oil testing equipment. The space and facilities used by official personnel must meet the specifications outlined below.

a. Location of Equipment.

The method used for determining the oil content of sunflower seed samples utilizes a magnet with predetermined electromagnetic strength to activate the hydrogen atoms present in the sample. Metal objects or a strong magnet placed near the NMR equipment may interfere with the electromagnetic field and produce erroneous results. Therefore, do not place metal objects adjacent to the NMR equipment. **Do not place the NMR instrument on a steel table**.

A vibration-free table should be used to support equipment. Also, when more than one electronic instrument is located in the same work area, maintain at least 60 centimeters (approximately 2 feet) distance between instruments.

In addition, NMR equipment must be placed in a location conducive to a stable environment and shielded from electrical or electromagnetic interferences. NMR equipment must be protected from drafts, heating and cooling vents or devices, and preferably be kept away from outside walls and windows.

b. Temperature.

Maintain a constant temperature between 18°C (65°F) and 30°C (85°F) in the room where the NMR instrument is located. Record room temperature using a calibrated thermometer located near the NMR instrument.

Insert the thermometer into a small glass or plastic bottle filled with sunflower seed to reduce erroneous readings. The depth of the sunflower seed must be sufficient to cover the insertion level of the thermometer.

Fluctuation in room or sample temperature adversely affects analysis results. After calibration, a change in room or sample temperature greater than 0.5°C will require re-calibration using the SSS. Therefore, locate the NMR equipment in a room where the temperature remains very stable to minimize the need for re-calibration.

c. Power Supply.

The power for NMR instruments shall be supplied by a 120 ± 10 VAC 15-20 amp dedicated circuit. A maximum of two electronic instruments and associated printers may be placed on one dedicated circuit (2 NMR or 1 NMR and 1 NIRT instrument). To reduce interference from other sources, do not place other equipment on the circuit.

d. Dust.

Accessible surfaces of the NMR instrument, balance, and surrounding area shall be maintained essentially free from contaminants. Use a vacuum cleaner and brush for maintaining a clean and dust-free environment in the sunflower seed testing area. **Do not** use compressed air for clean-up purposes.

At locations where a dust collection system is not available, place the NMR instrument and balance in a room separate from all dust-producing equipment such as grinders, dockage testers, and dividers.

## 2.5 INITIAL LABORATORY SETUP

Specified service points must observe certain guidelines when establishing new testing laboratories and/or placing new equipment in service.

a. New Laboratories.

Upon request, TSD will assist agencies in planning and preparing laboratories for official oil testing service. Agency managers must notify the field office manager that a new laboratory is being planned and provide a diagram of the proposed laboratory design. The diagram should contain the proposed locations of NMR oil testing equipment, location of major inspection equipment (e.g., dockage testers, dividers, etc.), and a description of the power supply. Any additional information regarding the laboratory setup or equipment should also be discussed.

The monitoring field office will forward a copy of all submitted information to the TSD for review. Upon receipt, the TSD will review the information and make recommendations to the agency and monitoring field office to facilitate the laboratory setup.

b. New Equipment.

Notify TSD before placing newly purchased NMR instruments in service. TSD will provide instructions to check the accuracy of the instrument and correct any deficiencies before the instrument is placed into official service. If problems are identified the checkout process may take several days to complete; therefore, contact TSD as soon as possible. Do not use newly purchased instruments for official NMR oil testing until the instrument has been checked and accepted by TSD.

# NMR Calibration/Check Sample Log

Date of Initial Calibration: _____ (pulsed NMR instruments only)

Low SSS #: _____ Low SSS RV: _____ High SSS #: _____ High SSS RV: _____

Tolerance: If the measured value exceeds the reference value (RV) by +/- 0.3, retest the SSS. If the measured value still exceeds the RV by +/- 0.3 re-standardize the instrument and retest the SSS.

| Date | Time | Temp. | Low SSS Value | Difference From RV | High SSS Value | Difference From RV | Temp. |
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# CHAPTER 3

# SAMPLE PREPARATION

## 3.1 BASIS OF DETERMINATION

Determine sunflower seed NMR oil on a representative portion cut from the sample after the removal of foreign material. Report the sunflower seed NMR oil percent on a 10 percent moisture basis (mb).

## 3.2 CLEANING SAMPLES

Use a Boerner divider to obtain a representative sample portion of approximately 50 to 55 grams. Mechanically clean the sample portion using a Carter Dockage Tester operated as specified in the Grain Inspection Handbook, Book II. Sunflower seeds and dehulled seeds that pass over the riddle and the material that passes over the No. 3 and over the No. 8 sieves are combined to form the mechanically cleaned sample. Handpick the mechanically cleaned sample portion to remove all matter other than sunflower seed and dehulled seeds.

## 3.3 PORTION SIZE

The sample used to determine the NMR oil must not extend above the top of the Radio Frequency (R.F.) coil. For a 150 ml NMR sample tube, a depth of 50.8 to 63.5 mm (2 to 2 1/2 inches) must be observed.

Before drying the handpicked sample portion, check the sample volume using a marked NMR sample tube. If the sample volume does not fall within the required range, adjust the sample size until it does. If the deviation is large, use a Boerner divider to adjust the sample size.

## 3.4 DRYING SAMPLES

The technique used to determine sunflower seed oil is based on measuring the number of electromagnetically activated hydrogen atoms in a sample. Therefore, liquid hydrogen atom sources other than oil, such as moisture, must be removed prior to NMR oil determination.

Perform the following procedures for drying the sunflower seed sample to remove moisture:

a. Preheat the moisture oven to 130°C and check the oven temperature with a calibrated thermometer. The oven temperature must be 130°C $\pm$ 2°C before drying samples.

b. Place the handpicked sunflower seed sample in a moisture dish and record the dish ID number.

c. Place the sample in the preheated moisture oven with the lid under the dish. Dry the sample for 3 hours. Begin timing the drying once the temperature reaches 130°C ± 2°C.

## 3.5 COOLING SAMPLES

After drying, the sunflower seed samples must be stabilized to room temperature before performing NMR oil determination. In addition, room and sample temperature must be within ± 0.5°C from the temperature recorded during restandardization.

Allow samples to cool to room temperature using one of the following procedures:

a. Desiccating Cabinet Method.

(1) Place a calibrated thermometer inside the desiccating cabinet.

(2) Immediately after removing the samples from the drying oven, place the lid on the dish and place the dish in the desiccating cabinet. Monitor the desiccating cabinet temperature.

(3) When the room and desiccating cabinet temperature are within ±0.5°C, samples are ready for NMR oil determination. The cooling time will be a minimum of 3 hours.

b. NMR Sample Tube Method.

A thermometer inserted through a rubber stopper is needed to measure the temperature of at least 1 sample in a rack (maximum 10 samples per rack) as they cool. The thermometer must be inserted into the rubber stopper so that approximately 25 mm of the thermometer will be immersed in the sunflower seed sample during the cooling period.

(1) Immediately after removing each sample from the drying oven, pour the sample into a marked NMR sample tube and seal with a rubber stopper. Each rack must have at least one rubber stopper with a thermometer inserted.

(2) Place the NMR sample tube into a rack. The rack should keep sample tubes separated by approximately 20 mm.

(3) When room temperature and the thermometer reading are within ± 0.5°C, the samples are ready for NMR oil determination. The cooling time will be at least 3 hours.

# CHAPTER 4

# CERTIFICATION

## 4.1 SEED TYPE DECLARATION

Applicants requesting sunflower seed oil determination must declare the seed type as part of the request for service. Written confirmation or a verbal declaration may be required at the discretion of official personnel.

Applicants may make blanket declarations for all lots or a specific type of service. For example, "All inbound lots of sunflower seed are of high-linoleic-type unless specified otherwise".

The official certificate shall include a qualifying statement reflecting applicant declaration (see section 4.3 c).

## 4.2 EXPORT LOTS

Applicants for service may request official sunflower seed oil determination on the basis of sublot samples or a composite sample. The type of service and the quantity of sunflower seed to be loaded must be indicated on the load order document.

a. Sublot Basis.

(1) Obtain a representative sample for each sublot.

(2) Follow the official procedure to determine NMR oil content for each sublot sample. Record the oil percentage on the inspection log to the nearest tenth percent.

(3) Using the standard FGIS rounding procedures, calculate the average NMR oil content for all sublots.

(4) Determine the highest and lowest NMR oil percent for all sublots.

(5) Report the high, low, and the average percent NMR oil (10% mb) to the nearest tenth percent on the certificate.

b. Ship Composite Basis.

(1) Based on the number of sublots being loaded, determine the amount of sample portion (in proportionate size) to be taken from each sublot that will provide a 1,000-gram composite sample.

(2) At the completion of ship loading, obtain a representative sample of at least 100 grams from the composite using a Boerner divider.

(3) Determine the NMR oil content using the official procedures.

(4) Report the percent NMR oil (10% mb), as determined on the composite sample, to the nearest tenth percent on the certificate.

## 4.3 CERTIFICATION

Record the percent oil of the sunflower seed using the appropriate approved statement in the "Remarks" section of the official grade certificate. Results may be reported on the same certificate with official grade or factor results or reported on a separate certificate.
When certifying oil alone (without official grade and factors), cross out the words "Grade and" and show only "sunflower seed" on the grade line of the certificate.

a. Approved Statements for Export Lots.

(1) Sublot Basis

"Oil content of sublots range from (lowest) % to (highest) %."

"Average oil content for all sublots ____ %, 10 percent moisture basis."

(2) Composite Basis

"Oil content ____ %, 10 percent moisture basis. Determination based on a composite sample."

b. Approved Statement for Domestic Lots.

"Oil content ____ %, 10 percent moisture basis."

c. Approved Qualifying Statements.

The applicant for service must declare the type of seed for NMR oil testing. Include one of the following statements on the certificate. Insert the appropriate sunflower seed (linoleic, mid-oleic, high-oleic) to complete the statement.

**Note: The term NuSun may be used in lieu of mid-oleic.**

"(_____) type sunflower seed, per applicant statement."

"Applicant states sunflower seed is (_____) type."

Field office managers can approve minor modifications to the statements provided the meaning and intent of the statements have not changed.

# CHAPTER 5

# MONITORING PROGRAM

## 5.1 GENERAL INFORMATION

The sunflower seed oil monitoring program is designed to monitor the accuracy of official sunflower seed oil determinations. Several methods are utilized to monitor particular elements in the sunflower seed oil testing process. These methods include local monitoring by the field office, TSD monthly check samples, intermarket sample exchanges, and special studies or collaboratives.

Monitoring information is used by field offices and inspection agencies for evaluating the performance of their local oil testing programs. TSD uses the information to evaluate the capability of the national sunflower seed oil testing program and the performance of the calibration standards.

Local monitoring by the field office and TSD check-sample results will identify service points having questionable oil results. Field offices and agencies must initiate follow-up and corrective action whenever oil testing problems are detected. Follow-up and corrective action includes investigating, identifying, and correcting oil problems. Intermarket sample exchanges, opinion samples, and special studies or collaboratives, are tools which are used to help identify the cause of discrepancies. TSD will assist the field in identifying the cause(s) of oil testing problems.

## 5.2 MONITORING FILE SAMPLES

Oil testing performance is evaluated through field office monitoring of official file samples. Sunflower seed oil program performance is determined by comparing original NMR oil results with monitoring results.

Specified service points are required to select and forward samples to the Grand Forks Field Office for monitoring. From the batch of monthly monitoring samples, the Grand Forks Field Office will select 10 samples and forward them to TSD for additional monitoring.

a. Selecting Samples.

Specified service points providing official NMR oil testing service shall use the following procedures to select monitoring samples.

(1) Select 10 sunflower seed samples per month representing the range of oil values observed during the month. **Do not** select all samples from the same day. When less than 10 samples are tested during a month, select all samples tested.

Service points that perform a low volume of official oil determinations may, with field office concurrence, periodically make up sets of 10 samples to check the accuracy of their instrument.

(2) Mail the samples to the Grand Forks Field Office by close of business on the last business day of the month.

**Note: The field office or TSD may request additional samples for monitoring purposes.**

b. Preparing Samples.

(1) Using a Boerner divider, obtain a portion weighing approximately 200 grams from each sample selected for monitoring. If a location frequently receives submitted samples of insufficient size to provide a 200-gram portion, the field office manager may approve a smaller monitor portion.

(2) Place each sample portion in a clear plastic bag and close securely.

(3) Include a completed monitoring data worksheet with the samples. Indicate any unusual conditions observed in the "remarks" section of the worksheet.

c. Packaging Samples.

(1) Place the selected sample portions and completed monitoring data worksheet in a canvas mailing bag.

(2) Prepare the appropriate mailing tag (business reply tag for delegated and designated agencies, metered tag for FGIS) by indicating "SF Oil Monitoring" on the reverse side and securely attach it to the bag.

d. Monitoring Results.

The field office will prepare control charts and transmit the sunflower seed oil monitoring results to the appropriate specified service points for their review and follow-up action.

e. Evaluating Results.

The field office and specified service point managers are responsible for evaluating the control charts. If control chart rule violations occur, they must initiate follow-up action, take the necessary corrective measures, and document any action taken to resolve differences between original and monitoring data.

Documentation may be placed directly on the control chart, in a ledger or notebook, and must indicate the action limit violation and the corrective action taken.

## 5.3 BI-MONTHLY CHECK SAMPLES AND SPECIAL STUDIES

Bi-monthly check samples issued by TSD are used to monitor the consistency of results from individual specified service points over a period of time, identify potential intermarket differences between service points, and to track the capability of the national oil testing program in relation to the reference method. Special studies are designed to resolve differences in oil results either within or between markets.

a. Bi-monthly Check Samples.

TSD will select bulk samples of sunflower seeds representing the range of oil values typically seen in the market and prepare multiple sets of representative portions. On a bi-monthly basis, a portion of each of these samples will be distributed to specified service points, FGIS field offices, and the TSD reference method lab to be tested for oil content. Participants must complete the oil testing within 7 working days of receipt of the samples and transmit the results to TSD.

TSD will tabulate, plot, and analyze the data and prepare a report of the average results of all locations compared to TSD. In addition, a chart of the historical results for each location will be prepared. Plots and statistical analysis will be transmitted to all participants and supervising FGIS offices.

Field office and specified service point managers must initiate corrective action and follow-up when needed. The TSD will assist the field offices in resolving intermarket differences and investigate and take necessary action when excessive differences between the NMR oil and reference methods are indicated.

b. Special Studies or Collaboratives.

Special studies or collaboratives, conducted at the discretion of TSD, are designed to resolve differences in NMR oil results within or between markets. Because special studies are normally of an urgent nature, an expedient resolution of the problem is essential. Therefore, all participants must perform the requested tests and report the results to TSD within 5 working days.

## 5.4 INTERMARKET SAMPLE EXCHANGE

An intermarket sample exchange is used to isolate oil differences between inspection points. Oil testing laboratories will determine oil results on separate portions obtained from the same sample. Oil results are then compared to determine whether significant differences between locations exist. This procedure is particularly useful when there are sunflower seed shipments between two specified service points or an individual applicant is routinely receiving service from two service points.

There are no restrictions as to which offices may exchange samples. Specified service points are encouraged to exchange samples with other service points and field offices for the purpose of investigating and resolving intermarket inspection differences. A copy of the results of the exchange must be provided to the field office and/or TSD for review.

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# CHAPTER 6

## INSTRUMENT SETUP AND SAMPLE ANALYSIS OXFORD 4000 CONTINUOUS WAVE NMR ANALYZER

## 6.1 INSTRUMENT SETUP

The operation of the OXFORD 4000 NMR analyzer is controlled by a microprocessor and instructions will appear on the display at each stage of the operation.

Operators must read the user=s manual and familiarize themselves with this instruction before operating the NMR instrument.

**NOTE: The OXFORD/FGIS EPROM program, which allows for a separate number of repeat parameters for calibration and market sample analysis, must be installed in order to use the instrument for official oil determinations.**

The oil values assigned to the SSS used in a two-point calibration are stated on a 10 percent moisture basis. As a result, the oil values reported by the instrument are also stated on a 10 percent moisture basis.

a. Instrument Setup.

After the NMR instrument power is turned on, it automatically carries out diagnostic tests on the instrument hardware. Allow the instrument to warm up for at least 2 hours. Once the NMR instrument is turned on, it may be left on. However, if the NMR instrument is turned off, a 2 hour warmup is required before calibration and/or sample analysis.

If an error message is displayed, consult the user=s manual.

All instructions must be answered by entering the requested information via the keypad and pressing the **"ENTER"** key. Press the "**YES"** or "**NO"** keys to answer questions.

(1) ENTER YEAR.

(2) ENTER MONTH.

(3) ENTER DAY.

(4) ENTER HOUR.

(5) ENTER MINUTE.

(6) INSERT TUNING SAMPLE. Insert the tuning sample (TS). The instrument will automatically perform the tuning function.

b. Programmed Mode Setup.

To prepare the instrument for a new programmed mode, the operator must key in and enter the secret code number for the instrument when the message "ENTER MODE NUMBER" appears on the display.

The instrument will print "PROGRAM MODE" and "DATE and TIME." Follow the instruction as it appears on the display and enter the requested information.

The following steps describe how to setup a programmed mode.

(1) **ENTER R.F. LEVEL**. Enter the FGIS approved R.F. level of **225**. Press the **"ENTER"** key.

(2) **ENTER A.F. GAIN**. Enter the FGIS approved A.F. gain of **500**. Press the **"ENTER"** key.

(3) **ENTER GATE WIDTH**. Enter the FGIS approved width of **1.5 gauss**. Press the **"ENTER"** key.

(4) **TEMPERATURE CONTROL REQUIRED?** Temperature control is not required. The NMR instrument will be re-calibrated before proceeding with NMR measurements if the sample or room temperature fluctuates more than **"** 0.5°C. Press the **"NO"** key.

(5) **MASS INPUT REQUIRED?** The dry sample weight is necessary to calculate the signal per gram ratio. Press the **"ENTER"** key.

(6) **TWO POINT CALIBRATION?** Two SSS with known oil and dry sample weights are required to determine the calibration slope. Press the **"YES"** key.

(7) **ENTER PERCENT CONCEN FOR REF XXA**. Enter the oil percent for the low value SSS. Press the **"ENTER"** key.

(8) **ENTER PERCENT CONCEN FOR REF XXB**. Enter the oil percent for the high value SSS. Press the **"ENTER"** key.

**NOTE: Mode numbers are sequentially assigned by the program. XX is the number assigned to this mode.**

(9) **ENTER ANALYSIS TIME**. Enter the FGIS approved analysis time of **32** seconds. Press the **"ENTER"** key.

(10) **ENTER ANALYSIS REPEATS**. Enter **1** for the number of measurements for official NMR oil testing (initial analysis plus one repeat). Press the **"ENTER"** key.

(11) **ENTER CALIBRATION TIME**. Enter the FGIS approved calibration time of **128** seconds. Press the **"ENTER"** key.

(12) **ENTER CALIBRATION REPEATS**. Enter **2** for the number of measurements for instrument calibration (initial analysis plus two repeats). Press the **"ENTER"** key. The printer will now list the selected parameters.

(13) **USE MODE?** Press the **"YES"** key to enter the analysis mode and try the selected parameters before permanently storing it. If the **"NO"** key is pressed then execution continues at the next question.

(14) **STORE MODE DETAILS?** Press the **"YES"** key to store the programmed mode parameters in the permanent memory. Programs stored in the permanent memory can be recalled and used at a future time by entering the designated code number. The operation will continue at step 16. To discard the program, press the **"NO"** key, and the operation will continue at step 15.

(15) **ARE YOU SURE?** Press the **"NO"** key to return to step 13 above or press the **"YES"** key to discard the program.

(16) This mode is now programmed and stored. Record the programmed mode number and parameters in the instrument record book.

(17) **ENTER MODE NUMBER**. Enter a programmed mode number to proceed to calibration and analysis.

c. Tuning the NMR Instrument.

The instrument will automatically perform the tuning function during setup and calibration. The operator may tune the NMR instrument at any time a signal separation appears on the oscilloscope by performing the following:

(1) Insert the TS in the magnet coil assembly.

(2) Press the tuning button located above the on/off switch on the back of the instrument.

(3) If necessary, repeat step 2 above until no signal separation appears on the oscilloscope.

## 6.2 INSTRUMENT CALIBRATION

a. Daily Calibration.

The NMR instrument must be calibrated using the SSS before testing market samples, when room or sample temperatures change by **"**0.5°C or more, and when the result for SSCS exceeds the established tolerance.

When using a mode for the first time, the instrument will automatically initiate the calibration function.

When changing modes to a previously calibrated mode, press **"YES"** to enter the calibration function when prompted to **"RE-STANDARDIZE."**

To re-calibrate while analyzing market samples, enter **"0"** for the sample ID and press **"ENTER."**

The following steps describe the calibration process:

(1) **RECORD ROOM TEMPERATURE**. Record to 0.1°C on the Daily Calibration/Check Sample Log.

(2) **INSERT TUNING SAMPLE**.

(3) **ENTER REF XXA MASS**. Enter the weight of the low value SSS. The weight of the sample contained in the SSS is recorded on the SSS label.

(4) **REF XXA INSERTED?** Insert the low value SSS into the magnet coil assembly. Press the **"YES"** key.

(5) **MEASUREMENT IN PROGRESS**. The measurement time, in seconds, will appear in the top of the display and will proceed to count down to zero. The measurements will be repeated as specified in the programmed mode.

(6) **ENTER REF XXB MASS**. Enter the weight of the high value SSS. The weight of the sample contained in the SSS is recorded on the SSS label.

(7) **REF XXB INSERTED?** Insert the high value SSS into the magnet coil assembly. Press the **"YES"** key.

(8) **MEASUREMENT IN PROGRESS**. The measurement time in seconds will appear in the top of the display and will proceed to count down to zero. The measurements will be repeated as specified in the programmed mode.

(9) After taking the last NMR reading, check the room temperature. Disregard and repeat the calibration if a change in room temperature of greater than "0.5°C has occurred.

(10) Examine the NMR responses. If the difference between the high and low NMR responses is 0.70 NMR units or less, test the SSS as a market sample. If the difference between the high and low NMR responses is greater than 0.70 NMR units, disregard the readings and repeat calibration. If the problem persists, contact the BAR.

b. Accuracy Check Using the SSS.

Test the SSS as a market sample to check the NMR instrument accuracy after daily re-standardization or when the room temperature changes by "0.5 ° C or after every 15-20 samples have been analyzed or hourly whichever comes first. Maintain a record (electronic or written) of the calibration checks using the Calibration/Check Sample Log as a template.

(1) Enter the SSS ID and seed weight. The instrument will perform two measurements and determine the oil content.

(2) When the analysis is complete examine the NMR responses. If the difference between the NMR responses is 0.60 NMR units or less, accept the results. Otherwise, disregard and repeat the measurements. Record the oil value on the Calibration/Check Sample Log.

(3) Record the SSS temperature to 0.1 ° C on the Calibration/Check Sample Log. Compare the current temperature reading to the temperature recorded at step 5.2 a.(1). Repeat the calibration and check sample test, if a change in temperature of "0.5 ° C has occurred.

(4) Calculate the difference between the SSS results obtained and the Reference Values (RV) and record this value on the Calibration/Check Sample Log. If the difference for either SSS exceeds "0.3 retest the SSS. If the difference still exceeds "0.3, re-calibrate the instrument and re-test the SSS.

## 6.3 ANALYZING MARKET SAMPLES

a. Testing Market Samples.

Once the instrument has been properly checked and calibrated, begin analyzing market samples as follows:

(1) **USE MODE XX?** Press the **"YES"** key to continue with the current mode or **"NO"** to use a different programmed mode. If you change modes, you will be required to calibrate the new mode before using it.

(2) **ENTER SAMPLE NUMBER**. Key in the sample identification. Press the **"ENTER"** key.

(3) **ENTER SAMPLE XXXXX MASS**. Key in the sample weight (dry weight). Press the **"ENTER"** key.

**NOTE: The instrument will also accept sample weight directly from an electronic balance connected to the RS-232 port. If a balance is connected, place an empty moisture tin on the balance and zero the balance. Pour the dry sample into the bin. After the balance reading reaches a constant value, press the "YES" key on the NMR instrument to enter the sample weight.**

(4) **SAMPLE XXXXX INSERTED?** Transfer the dry sample to a 150 ml NMR sample tube and insert it into the magnet coil assembly. Press the **"YES"** key.

(5) **EXAMINE NMR RESPONSES**.

(a) The instrument will print the sample ID, date, time, NMR response for each of the two measurements, mean NMR response, signal mass ratio, and NMR oil percent on a 10 percent moisture basis.

(b) If the difference between the two NMR responses is 0.60 units or less, accept the results.

(c) If the difference exceeds 0.60 units, disregard the results and repeat the analysis of the sample. If the problem persists, re-calibrate the NMR instrument using the SSS.

(d) If the range of measurements between NMR responses is continually above 0.60 units, contact TSD.

b. Reporting Results.

Record and report the percent oil on the pan ticket, inspection log, and certificate to the nearest tenth percent using the standard FGIS rounding procedures.

## 6.4 INSTRUMENT DIAGNOSTIC

If an NMR instrument is functioning properly, less than 5.0 percent of the results should be rejected because the difference between the high and low NMR responses exceeds 0.60 or 0.70 NMR unit tolerance for two and three-cycle analyses, respectively. A significantly higher rate of rejected analyses indicates a problem with the NMR instrument.

a. Eratic NMR Response.

The most common cause of erratic responses is a poor connection between the coil and the console. If tightening the connector does not eliminate the problem, disconnect the cable, clean the contacts with a clean pencil eraser, and reconnect. If the problem still persists, contact Oxford technical support.

b. Distorted or Weak Oscilloscope Signal.

A distorted or flickering oscilloscope signal is generally due to a poor connection between the coil and the console. Check the connector at the console and tighten if necessary. If tightening the connector does not eliminate the problem, disconnect the cable, clean the contacts with a clean pencil eraser, and reconnect. If the problem still persists, contact Oxford technical support.

A weak or dim oscilloscope signal may indicate that the cathode ray tube is near failure. When the instrument is left on but not in use, the brightness control should be turned to the lowest level to extend the life of the cathode ray tube.

c. Consistently High or Low Results.

Consistently high results are generally due to incomplete drying of the sample. If incompletely dried seeds are measured, the NMR signal will include the total amount of water and oil present and will give an inflated indication of oil content.

High results can also occur because of differences in temperature between the SSS at the time of calibration and the market sample. If the market samples are significantly cooler than the temperature of the SSS at the time of calibration, the indicated oil content will be inflated.

Consistently low results are commonly caused by not allowing the market samples to cool to room temperature. If the market samples are significantly warmer than the temperature of the SSS at the time of calibration, the predicted oil content will be lower than the actual oil content.

Low results can also be caused by contamination by ferrous materials. Small amounts of ferrous materials present in samples will reduce the signal extensively.

# CHAPTER 7

## INSTRUMENT SETUP AND SAMPLE ANALYSIS - OXFORD MQA 6005 AND MQA 7005 PULSED NMR ANALYZERS

## 7.1 INSTRUMENT SETUP

a. Setup Information.

Operators must read the user's manual and familiarize themselves with this instruction before operating the NMR instrument. The operation of the OXFORD MQA 6005 and MQA 7005 pulsed NMR instruments is controlled by a personal computer. After the instrument is powered on, a series of automatic internal tests are carried out to confirm proper instrument functionality. Any error messages should be referenced in the Operator's manual.

The magnet is maintained at a temperature of 40 Degrees Centigrade. The unit must not be used for a period of 6 hours after being switched on to allow the magnet temperature to stabilize. After being switched-on the instrument should remain on.

b. Activation Of Undried Sample Oil Conversion Software.

Upon turning on the instrument, one of the icons displayed is "SHORTCUT TO SUNFLR1." Using the "mouse" activate the icon and a conversion window will appear on the screen. Again using the "mouse," activate the "-" button located in the upper right corner of the window. This will shrink the window into a "MQA OIL CORRECTION" button located on the lower task bar of the screen. This button is activated by the operator each time the instrument is used to measure the oil content of undried samples.

This setup procedure for activating the undried sample oil conversion software must be repeated each time the power is turned on.

## 7.2 INITIAL INSTRUMENT CALIBRATION

The NMR instrument must be initially calibrated, using the GIPSA Sunflower Seed Standards (SSS). Once the instrument has been calibrated, the instrument is re-standardized (drift corrected) daily using the SSS or when the room temperature changes by $\pm$ 1° C.

Follow the procedures listed below for calibrating instruments.

a. Select "GIPSA Oil in Dried Seeds" method from the MQA Methods menu.

b. Select Optimize (F8) from the Oxford MQA Methods main menu to activate the Optimize screen.

c. Select **Frequency** and **Pulse Times** using the check boxes.

d. Click on "Go" (green triangle) or press F5.

e. At the prompt, insert the Oxford Test Sample (mineral oil tuning sample) and click on "Go" (green triangle) or press F5 to start the frequency optimization.

f. At the prompt, click on "Go" (green triangle) or press F5 to start the pulse times optimization.

g. If no error messages are displayed, click on "Accept" (green check) or press F2 to validate the optimization. Remove the test sample.

**NOTE: If an error message is displayed, contact Oxford technical support.**

h. Select "GIPSA Oil in Dried Seeds" method from the MQA Methods menu.

i. Select "Calibrate" or press F7 from the Oxford MQA Methods main menu to display the calibration editor.

j. In the **Data set** panel then double click on the "CALMM/DD/YYYY" **Data set.**

k. Rename the Data set by replacing the "MM/DD/YYYY" with the current date.

**NOTE: The "CALMM/DD/YYYY" data set should be the only data set present on a new instrument. Additional calibration data sets can be added by clicking on the blue plus (+) button. Name new calibrations using the "CALMMDDYYYY" format.**

l. Click on "Accept" (green check) or press F2 to exit the **Data set** editor.

m. Note the SSS temperature to 0.1° C.

n. Click on "Go" (green triangle) or press F5 to start measurement for the first calibration standard.

o. Insert the Low SSS calibration standard into the instrument.

p. When the measurement is complete, remove the SSS from the magnet.

q. Enter the SSS ID, SSS Mass and Reference Oil value from the SSS label. During this process the point appears on the calibration graph, flashing inside a gray dashed box.

r. Check the "Calib. Standard" box and the "Setup Standard" box.

s. Click "Accept" (green check) or press F2 to validate the acquisition of this point.

**NOTE: If Reject (F3) is selected, the point will be lost. If the reference value for either component is left as zero then the software will ask for confirmation.**

t. Click on "Go" (green triangle) or press F5 to start measurement of the second calibration standard.

u. Insert the High SSS calibration standard into the instrument and repeat steps p through s listed above.

**NOTE: Once a minimum of two calibration standards has been measured, a solid calibration line is shown.**

v. Click "Accept" (green check) or press F2 to exit the calibration program.

## 7.3 DAILY INSTRUMENT RESTANDARDIZATION

a. Daily Re-standardization.

When the instrument is first put into service, it must be calibrated following the procedures in section 7.2. Once the calibration has been established, the following procedures are used daily to optimize the instrument and correct for drift due to temperature changes.

(1) From the main menu, select the "GIPSA Oil in Dried Seeds" method.

**NOTE: If only one method is installed, it will be automatically selected.**

(2) Select "Optimize" (F8) from the Oxford MQA Methods main menu to activate the Optimize screen.

(3) Select **"Frequency"**, **"Pulse Times"** and **"Re-standardization"** (check boxes).

(4) Click on "Go" (green triangle) or press F5 and click "OK" to confirm that you wish to re-standardize.

(5) Record the SSS temperature to 0.1° C on the Check Sample Log.

(6) When prompted, insert the Oxford Test Sample (mineral oil tuning sample) and click on "Go" (green triangle) or press F5 to optimize the resonance frequency.

(7) When the frequency optimization is complete, click on "Go" (green triangle) or press F5 again to optimize the pulse times.

(8) When prompted, remove the Oxford Test Sample, insert the Low SSS, and click on "Go" (green triangle) or press F5.

(9) When prompted, remove the Low SSS, insert the High SSS, and click on "Go" (green triangle) or press F5.

(10) If no error messages are displayed, click on "Accept" (green check) or press F2, and remove the High SSS. If an error message is displayed, contact Oxford technical support.

b. Calibration Check Using the SSS.

Test the SSS as a market sample to check the NMR instrument accuracy after daily re-standardization or when the room temperature changes by $\pm$ 1° C or after every 30-40 samples have been analyzed or hourly, whichever comes first. Maintain a record (electronic or written) of the calibration checks using the Calibration/Check Sample Log as a template.

(1) From the Oxford MQA Methods menu, select "GIPSA Oil in Dried Seeds" method, then "Analyze" (F5) to display the routine analysis screen.

(2) Click on "Go" (green triangle) or press F5 and insert the Low SSS.

(3) Enter the SSS ID and mass.

(4) When the analysis is complete remove the SSS from the magnet, record the reference value on the Daily Calibration Check Log, and click on "Accept" (green check) or press F2 to validate the result.

(5) Click on "Go" (green triangle) or press F5 and insert the High SSS.

(6) Enter the SSS ID and mass.

(7) When the analysis is complete remove the SSS from the magnet, record the reference value on the Daily Calibration Check Log, and click on "Accept" (green check) or press F2 to validate the result.

(8) Record the SSS temperature to 0.1° C on the Check Sample Log. Compare the current temperature reading to the temperature recorded at step a (5). Repeat the re-standardization and check sample test, if a change in temperature of ± 1° C has occurred.

(9) Calculate the difference between the SSS results obtained and the Reference Values (RV) and record this value on the NMR Check Sample Log. If the difference for either SSS exceeds ± 0.3 retest the SSS. If the difference still exceeds ± 0.3, re-standardize the instrument and re-test the SSS.

**NOTE: If the values repeatedly exceed the tolerances, you may need to re-calibrate the instrument. Contact TSD for technical support.**

## 7.4 ANALYZING MARKET SAMPLES

a. Testing Market Samples.

After the instrument has been re-standardized, analyze market samples as follows:

(1) If you are already in analysis mode, go to step 2. If not, from the Oxford MQA Methods menu, select "GIPSA Oil in Dried Seeds" method, then "Analyze" (F5) to display the routine analysis screen.

(2) Click on "Go" (green triangle) or press F5 and insert the sample into the magnet assembly.

(3) Enter the sample identifier (ID) and mass while the sample is being analyzed.

**NOTE: For users employing a Mettler balance and using the automatic sample weight transfer option only the sample identifier is entered.**

The procedures required for using the automatic sample weight transfer option are as follows:

(a) The MQA-6005/7005 must have the enable external balance activated.

(b) Prior to making a set of method measurements remove the sample cup from the scale and tare. Observe that the lower weight display is approximately 0.000.

(c) Place the empty sample cup on the scale and wait until the weight display shows the cup tare weight.

(d) Fill the sample cup with the sample from the sample cell and wait until the weight display shows the sample weight in red.

(e) Remove the sample cup from the scale, refill the sample cell, and measure the oil content of the sample using the MQA-6005/7005. When the sample cell is inserted into the magnetic module the weight will automatically be entered. Repeat steps (c) through (e) for additional samples.

**NOTE: During analysis, interim results are returned after the mass has been entered. Those interim results appear in white. When the analysis is complete the final result appears in black. In the event of a data entry error, the mass value can still be re-entered at this stage.**

(f) For undried samples, press the "MQA OIL CORRECTION" button on the computer lower left display task bar and a conversion window will appear. Enter the MQA-6005 or MQA-7005 oil and GAC moisture, and the percent oil at 10% moisture is automatically displayed. Press the "Enter" button to store the data in a text file (c:\qt\reports\sunflwr.txt). After recording the oil at 10% moisture, press the upper right corner "-" button to make the conversion window disappear and to reset the conversion software.

**NOTE: After testing a sample for moisture content with the GAC-2100 instrument, place the sample in a closed container until oil testing is performed. This will minimize changes in moisture content of the sample.**

**NOTE: If the GAC-2100 moisture result is not measured within 24 hours prior to the oil measurement, or the GAC-2100 moisture result is not between 4.5% and 10.5%, the undried sample oil measurement procedure cannot be used. Samples that do not qualify for the undried oil measurement procedure must be tested after drying (air-oven method).**

(4) When the measurement is complete, click on "Accept" (green check) or press F2 to validate the result. To cancel the measurement, click on "Reject" (red X) or press F3.

(5) To repeat the analysis of a single sample, you must remove the sample from the magnet and allow it to equalize to room temperature (10-15 minutes). Once the sample has returned to room temperature repeat steps (b) to (d).

**NOTE: Once the result has been accepted it is added to the report file and added to the trend graph. The trend plot can be viewed by clicking on the Trend Graph tab. The data is also sent to a temporary print file, which will print when a page of data has accumulated or when you exit the program.**

b. Reporting Results.

Record and report the percent oil on the pan ticket, inspection log, and certify to the nearest tenth percent using the standard FGIS rounding procedures.

# CHAPTER 8

## INSTRUMENT SETUP AND SAMPLE ANALYSIS - RESONANCE MARAN ULTRA PULSED WAVE NMR ANALYZER

## 8.1 INSTRUMENT SETUP

a. Setup Information.

Operators must read the user's manual and familiarize themselves with this instruction before operating the NMR instrument. Any error messages should be referenced in the Operator's manual.

The operation of the Resonance MARAN Ultra instrument is controlled by a personal computer. After the instrument is powered on, a series of automatic internal tests are carried out to confirm the instrument communications and the functionality of the console unit.

The internal magnet is maintained at a temperature of 40 Degrees Centigrade. The unit must not be used for a period of 12 hours after being switched on to allow the magnet temperature to stabilize. After being switched-on the instrument should remain on.

(1) Open the desktop folder "EasyCal Applications."

(2) Start the "EasyCal for Oilseeds" software by double-clicking on the icon.

**NOTE: Alternatively, select the icon with a left mouse click, followed by a right mouse click and selecting the "Open" option in the pop-up menu.**

(3) Enter the administrative password to get access to the calibration software and click "OK" to enter the software. "MARAN" is the password installed at the factory.

(4) Insert the tuning sample (100 grams of vegetable oil) to begin the parameter optimization and verification of instrument performance. The instrument will optimize the instrument parameters in the order of resonance frequency, pulse width, and instrument sensitivity. Once the software has completed one cycle of these tests it will perform the same tests a second time.

(5) When the second cycle of tests is completed, record the operating parameters: Frequency Offset (Auto Tuning or 01), RF Pulse Width (Autoset P90), and Receiver Gain (Autoset RG) on the calibration log (Attachment A).

If the difference between the new values for P90 and RG and the previous results are greater than 5 percent of the previous reading, the user should stop the setup process and contact Resonance for technical support.

**NOTE: Clicking on the "Results" button will display a comparison of the current and previous parameters.**

(6) Click "Continue" to begin the calibration procedure.

b. Calibration.

(1) "GIPSA Dry Seed" for the name of the calibration and click "OK".

(2) Enter the sample ID, weight, and oil content of the High Sunflower Seed Standard (SSS) and click the "Continue" button.

(3) Insert the High SSS.

**NOTE: The instrument sensitivity will be optimized base on the sample highest in oil content. During the first part of the measurement, "EasyCal" maximizes the receiver gain (RG) parameter (adjustable from 0 - 100%) to a maximum value. Once the receiver sensitivity is optimized, the NMR signal is recorded for the high standard.**

(4) When the measurement is complete, remove the High SSS.

(5) Click "Yes" to measure the second standard.

(6) Enter the sample ID, weight, and oil content of the Low SSS and click the "Continue" button.

(7) Insert the Low SSS.

(8) Once the calibration standard has been measured, click "No" and remove the Low SSS from the magnet.

(9) Select the "Automatic" calibration method option. The two measured points are plotted as NMR signal/gram of seeds versus oil content, along with the statistics of the calibration. Record the value intercept, concentration intercept, and slope on the calibration log.

(10) Click "Yes" to accept the calibration.

**NOTE: If prompted to confirm overwriting of the existing calibration, click yes.**

(11) Click on the "Exit" button to leave the calibration program.

(12) Click "Yes" to confirm that you wish to terminate the "EasyCal Calibration" program.

c. Calibration Check.

Check the calibration by testing the SSS as market samples before beginning routine sample analysis.

(1) Open the desktop folder titled "EasyCal Calibrations" by double clicking on it.

**NOTE: All calibrations generated using the"EasyCal" software will be found in this folder.**

(2) Double click on the "GIPSA Dry Seed" calibration icon.

**NOTE: Before measuring seeds, the instrument will perform tuning and diagnostics to verify the instrument is performing within specifications.**

(3) Insert the tuning sample (100 grams of vegetable oil sample) in the instrument to begin the automatic tuning procedure.

**NOTE: The tuning procedure consists of first checking the instrument frequency. Once the 4 scan sequence is completed, the software will display a window of the old value, new value, and the difference. The software will then automatically proceed to measure the signal intensity for the tuning sample. The software will record the signal intensity in a diagnostic file. These values are recorded in a running file MQDiagnosis.log in the following directory**

**C:\Program\Files\Resonance\RlCalib\bin**

(4) Record the new tuning frequency value (01) on the check sample log and click "Yes" to accept the new value.

(5) Remove the tuning sample and click on the "Start Analysis" button.

(6) Enter the ID and the weight of the High Sunflower Seed Standard (SSS) and click "OK".

(7) When prompted, insert the High SSS.

(8) When the analysis is complete, remove the SSS, record the oil value on the check sample log, and calculate the difference between the reported value for the SSS and the known value. If the difference is greater than 0.3, repeat the analysis. If the repeat analysis result difference is still greater than 0.3, recalibrate the instrument.

(9) Repeat steps 6 - 8 with the Low SSS.

**NOTE: Test the SSS as a market sample to check the NMR instrument accuracy after calibration, when the room temperature changes by ± 0.5° C, after every 30 - 40 samples have been analyzed, or every two hours, whichever comes first. Maintain a record (electronic or written) of the calibration checks using the Check Sample Log in Appendix B as a template.**

(10) After completing the check sample procedure, follow the procedures listed below to begin analyzing market samples.

## 8.2 SAMPLE ANALYSIS

a. Analyzing Samples.

For each new sample, a window for entering the sample details will be presented. Follow the steps listed below for analyzing a sample.

(1) Enter the sample mass.

**NOTE: The sample mass can be entered manually, or if an electronic scale is connected, via the weigh button.**

(2) Enter the Sample ID and click "OK" to proceed.

(3) Insert the sample in the instrument. The instrument will display the oil content when the analysis is completed.

**NOTE: The sample ID, oil content, time and date of analysis will be logged to a log file. All log files are located in the following subdirectory:**

**C:\Program Files\Resonance\RlCalib\bin**

(4) Repeat steps (1) - (3) for each sample analyzed. Once the last sample is analyzed and removed from the analyzer, click the "CANCEL" button and then select "QUIT".

(5) Click "Yes" to terminate the "Rl Analysis" Program.

b. Reporting Results.

Record and report the percent oil on the pan ticket, inspection log, and certify to the nearest tenth percent using the standard FGIS rounding procedures.

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# CHAPTER 9

## INSTRUMENT SETUP AND SAMPLE ANALYSIS - BRUKER MINISPEC 7.5 PULSED NMR ANALYZER

## 9.1 INSTRUMENT SETUP

a. Setup Information.

After the instrument is powered on, a series of tests are carried out to confirm the instrument communications and the functionality of the console unit. The magnet is controlled at a temperature of 35.5 degrees Centigrade. The unit must not be used until two green lights are illuminated on the front of the magnet. This may take up to three hours.

(1) Start the minispec software on the personal computer.

(2) At the start of the day, select the "Daily Check" icon and run the auto-validation procedure.

(3) Insert the tuning sample (100 grams of vegetable oil). After the tuning sample is inserted the instrument will perform a series of tests.

(4) If no error message appears the instrument is ready for calibration.

(5) If an error message appears, select "Update Settings" from the minispec menu.

(6) Click "OK" to proceed to update all instrument settings.

(7) After the instrument updates the settings repeat the Daily Check. If the instrument repeatedly fails the Daily Check, contact technical support at Bruker.

b. Calibration.

The NMR instrument must be calibrated, using SSS, before testing market samples and when room or sample temperature changes by $\pm$ 0.5° C or more.

**NOTE: To recalibrate while analyzing market samples, at the prompt to enter or transfer from the balance the next sample weight, press the <ESC> key or with the mouse click the <STOP> button at the bottom of the screen. This terminates the <MEASURE> loop. With the mouse, select the <CALIBRATE> icon at the bottom of the screen or press ALT + C.**

The following steps describe the calibration process:

(1) Highlight the most recent calibration in the quick select list and select duplicate application from the file menu.

(2) Save a copy of the file using the following file name format (calMMDDYY) where "MM" is the month, "DD" is the day, and "YY" is the year.

(3) Select "Open" from the file menu and open the file created in step (2) above.

(4) Click on the "Calibrate" button.

(5) Enter the calibration name (calMMDDYY) for the result box title and click "OK".

(6) Enter the weight of the low value SSS and click "OK".

(7) Enter the oil content of the low value SSS and click "OK".

**NOTE: The weight and percent oil content of the seed contained in the SSS are recorded on the SSS label.**

(8) Insert the low value SSS into the sample compartment and click "OK".

(9) A visual progress thermometer and digital countdown of the number of scans will appear in the bottom of the program window and will proceed to count down to 0. Only one measurement will be required.

(10) When the analysis is complete, remove the SSS.

(11) Repeat steps (6) - (10) for the high value SSS.

(12) Click "Cancel" when prompted for data on sample 3.

(13) The instrument will display the calibration statistics. Record the slope and intercept values on the calibration log.

(14) Click the "Continue" button, then click "OK" to print and terminate the calibration process.

(15) Record room temperature to 0.1° C on the calibration log.

c. Calibration Check.

Check the calibration by testing the SSS as market samples before beginning routine sample analysis.

**NOTE: The minispec software allows the user to select from several sample data entry interfaces. The following instructions are based on using the "spreadsheet" interface for entering sample data. To configure the application to use the spreadsheet mode click on the "Configuration Table" icon (top of screen). If the "File Name" input box opens, select the configure application. Check the "Database Table Mode" in the option table.**

(1) Clear any previous data from the spreadsheet by highlighting the data and pressing the "Delete" key.

(2) Enter the ID and weight of the Low Sunflower Seed Standard (SSS) into the appropriate columns of the spreadsheet.

(3) Repeat step (2) for the High SSS.

(4) Use the mouse to select the <Measure> button.

(5) Insert the Low SSS and click "OK".

(6) When the measurement is complete (16 seconds), remove the Low SSS.

(7) Insert the High SSS and click "OK".

(8) When the analysis is complete remove the High SSS.

(9) Record the oil values on the Calibration/Check Sample Log and calculate the difference between the reported and known values for the SSS. If the difference is greater than 0.3%, repeat the analysis. If the repeat analysis result difference is still greater than 0.3%, recalibrate the instrument.

**NOTE: Test the SSS as a market sample to check the NMR instrument accuracy after calibration, when the room temperature changes by ± 0.5° C, after every 30 - 40 samples have been analyzed, or every two hours, whichever comes first. Maintain a record (electronic or written) of the calibration checks using the Check Sample Log in Appendix A as a template.**

## 9.2 SAMPLE ANALYSIS

Once the instrument has been properly validated (with the Daily Check procedure) and the instrument is calibrated, begin analyzing market samples

**NOTE: The minispec software allows the user to select from several sample data entry interfaces. The following instructions are based on using the "spreadsheet" interface for entering sample data. To configure the application to use the spreadsheet mode click on the "Configuration Table" icon (top of screen). If the "File Name" input box opens, select the configure application. Check the "Database Table Mode" in the option table.**

b. Analyzing Samples.

(1) Clear any previous data from the spreadsheet by highlighting the data and pressing the "Delete" key.

(2) Enter the ID's and weights of all of the samples to be tested into the appropriate columns of the spreadsheet.

**NOTE: The instrument will also accept weight directly from an electronic balance connected to the RS-232 port. If a balance is connected, place an empty container on the balance and zero the balance. Pour the dry sample into the tin and press the <PRINT> key on the balance. The sample weight will be transferred automatically as soon as the weight stabilizes.**

(3) Use the mouse to select the "Measure" button.

(4) Transfer the first sample to a 150-ml NMR sample tube, insert into the magnet, and click "OK".

(5) When the measurement is complete, remove the sample from the magnet.

(6) Repeat steps (4) and (5) for the remaining samples.

**NOTE: The data will automatically be save to a Microsoft Access database. The program will also print the date and time, sample ID, sample weight, and the NMR percent oil to an attached printer.**

b. Reporting Results.

Record and report the percent oil on the pan ticket, inspection log, and certify to the nearest tenth percent using the standard FGIS rounding procedures.